EPA Reviewer: William H. Donovan, Date 11/30/01

STUDY TYPE: Crop Field Trials - Bushberry subgroup; OPPTS 860.1500

TEST MATERIAL: Pyriproxyfen

FORMULATIONS AND TYPES: ESTEEM® 0.86 EC

SYNONYMS: None reported

CITATION: 455224-01 Samoil, K. (2001) Pyriproxyfen: Magnitude of the Residue on Blueberry: Laboratory Identification Number: 07233-99-MIR08. Unpublished study prepared by Interregional Research Project No. 4. 137 p.{OPPTS 860.1500}

SPONSOR: Interregional Research Project No. 4 (IR-4)

EXECUTIVE SUMMARY:

The results from eight crop field trials (MRID 455244-01) conducted on blueberry during 1999 have shown that the maximum pyriproxyfen residue was 0.64 ppm. Mature blueberry samples were collected 7 days after the last of two applications of pyriproxyfen (ESTEEM® 0.86 EC) at 0.097-0.102 lb ai/A (0.197-0.202 lb ai/A/season). A tolerance of 1.0 ppm is recommended to cover residues of pyriproxyfen in/on the bushberry subgroup, lingonberry, juneberry, and salal. As there are no Mexican, Canadian or Codex MRLs established for pyriproxyfen in/on blueberry, there are no compatibility issues to be reconciled.

The crop field trials for pyriproxyfen on blueberry are classified acceptable and satisfy the guideline requirement for crop field trials (Residue Chemistry Guidelines OPPTS 860.1500).

COMPLIANCE: Signed and dated GLP, Quality Assurance and Data Confidentiality were provided. The petitioner stated that some supporting data were not collected in strict adherence with 40 CFR 160 guidelines. However, these deviations do not affect the quality or integrity of the conclusions.

I. MATERIALS AND METHODS

The petitioner is proposing the GC/NPD Method RM-33P-1-3a as a tolerance enforcement method for residues of pyriproxyfen in/on the bushberry subgroup. A brief description and procedural recovery data were submitted in conjunction with the subject residue field trial data. The method, a version of Method RM-33P-1-3, is amended to include only pyriproxyfen analysis. Method RM-33P-1-3 has undergone an adequate radiovalidation, ILV trial, and PMV trial, and has been forwarded to the FDA for inclusion in PAM Vol. II (DP Barcode D257337, W. Donovan, 7/1/99). Samples of blueberry were analyzed at the National Food Safety and Toxicology Center, Michigan State University, (East Lansing, MI) using minor modifications.

Briefly, residues of pyriproxyfen in/on blueberry are extracted with acetone, filtered, and

concentrated. The residues are sequentially partitioned into acetonitrile:hexane and dichloromethane:0.5% NaCl solution, and cleaned-up using silica gel chromatography. Pyriproxyfen residues are then analyzed by GC/NPD and an external standard. The LOQ is 0.020 ppm in/on blueberry; the LOD was not reported.

For method validation, triplicate control samples of blueberry were fortified with pyriproxyfen at 0.023, 0.23, or 2.1 ppm. Recoveries in the respective ranges were 122-149%, 101-115%, and 100-108% (average ± SD, 115 ± 17%, n = 9). Apparent residues in/on untreated samples of blueberry fruit were all <0.02 ppm (<LOQ). Sample calculations and chromatograms were submitted.

The proposed GC/NPD Method RM-33P-1-3a is adequate for enforcement of tolerances for residues of pyriproxyfen in/on blueberry.

1. Test Compound

Chemical name: Pyriproxyfen
IUPAC: 4-phenoxyphenyl-(RS)-2-(2-pyridyloxy)propyl ether
CAS name: 2-[1-Methyl-2-(4-phenoxyphenoxy)ethoxy]pyridine
CAS #: 95737-68-1
Common name (ANSI, BSI or ISO): Pyriproxyfen
Developmental (Company) name: None reported

2. Trial Numbers and Locations

Crop	US Growing Regions									Total Trials
	1	2	5	12						
Blueberry										
Submitted	1	3	3	1						8
Requested	1	3	3	1						8

Comments: HED notes that the three trials in Region 5 were all conducted at one location: Fennville, MI. In addition, two of the three trials in Region 2 were conducted at a single location: Castle Hayne, NC. Because the applications were made using separate tank mixes on different days, HED is willing to consider these to be separate field trials for the use of pyriproxyfen in/on blueberries only. The limited blueberry growing regions, reduced risk status of pyriproxyfen, and the relative consistency of the residue levels found in the trials all contribute to this decision. Thus, geographic representation and the number of field trials conducted on blueberry are adequate.

3. Proposed Label Use Pattern

0.86 lb/gal EC

Crop	Application						Comments/ Restrictions
	Method/ Timing	Rate (lb ai/A)	Maximum Number	RTI[a] (days)	Total Seasonal Rate (lb ai/A)	PHI[b] (days)	
Bushberry[c]	Foliar	0.1	2	14	0.2	7	REI=12 hours. Do not apply through irrigation equipment.

[a] RTI = Minimum retreatment interval.

[b] PHI = Pre-harvest interval, the number of days between the last application and harvest.

[c] Bushberry crop subgroup includes blueberry (highbush, lowbush), currant, elderberry, gooseberry, huckleberry, lingonberry, juneberry, and salal ["Reviewer's Guide and Summary of HED ChemSAC Approvals for Amending Commodity Definitions [40 CFR § 180.1(h)] and Crop Group/Subgroups [40 CFR § 180.41]", B. Schneider, 20-SEP-2001].

Comments: A minimum volume for ground and air applications must be provided.

4. Analytical Method Validation (Concurrent)

Crop matrix	Analyte	Spiking Levels (ppm)	Recoveries obtained (%)	Range (%)	Mean ± SD (%)
Blueberry	pyriproxyfen	0.023; 0.046; 0.23	89-123; 122; 79-118	79-123	103 ± 14 (n=19)

Comments: Most recoveries were within the acceptable 70-120% range; however, some recoveries > 120% were noted in sample spiked at the lowest level (0.023 ppm). Based on the concurrent method recoveries, the GC/NPD method (Method RM-33P-1-3a) is adequate for collecting data on residues of pyriproxyfen in/on blueberry.

5. Storage Stability Conditions

Commodity	Active ingredient	Storage Temperature (°C)	Duration (days)
Blueberry	Pyriproxyfen	-25 to -16	73 and 78

Comments:
The maximum frozen storage interval from collection to analysis was 54 days for samples from the blueberry field trials. Frozen storage stability of pyriproxyfen residues was evaluated in conjunction with the blueberry field trials. Blueberry samples fortified with pyriproxyfen at 0.23 ppm and stored frozen (-25 to -16 C) for 73 and 78 days had recoveries of 58-89%, and 80-82%, respectively, providing no evidence of substantial degradation. Therefore, the residue values do not need to be corrected to account for degradation during storage.

Adequate storage stability data are available to support the residue data submitted for the permanent tolerance petition for the bushberry subgroup.

6. Application and RAC Information

Pyriproxyfen-ESTEEM 0.86 lb/gal EC-Blueberry

LOCATION (city /state)/YEAR	EPA Region	CROP/ VARIETY	APPLICATION[a]							HARVEST PROCEDURES			
			Growth Stage at first appl.	# of appl.	Interval (days)	Single Rate (lb ai/A)	Formulation	Method	Spray Volume (gal/A)	Growth Stage/ Harvest	Harvested Portion	Method/ equipment	Number and wt. of samples
Jonesboro/ ME/ 1999	1	Blueberry / Wild lowbush	fruiting	2	15	0.098-0.099	EC	Foliar broadcast spray	25	Mature	Fruit	Rake	Not Provided
Fennville/MI/ 1999	5	Blueberry / Rubel	fruiting	2	13	0.100-0.102	EC	Foliar directed (airblast) spray	50-51	Mature	Fruit	Hand	Not Provided
Fennville/MI/ 1999	5	Blueberry / Rubel	fruiting	2	13	0.100-0.102	EC	Foliar directed (airblast) spray	50	Mature	Fruit	Hand	Not Provided
Fennville/MI/ 1999	5	Blueberry / Rubel	fruiting	2	14	0.099-0.100	EC	Foliar directed (airblast) spray	50	Mature	Fruit	Hand	Not Provided
Castle Hayne/ NC/ 1999	2	Blueberry / Croatan	fruiting	2	14	0.099-0.100	EC	Foliar directed spray	28	Mature	Fruit	Hand	Not Provided
Castle Hayne/ NC/ 1999	2	Blueberry / Croatan	fruiting	2	14	0.099	EC	Foliar directed spray	28	Mature	Fruit	Hand	Not Provided
Chatsworth/ NJ/ 1999	2	Blueberry / Bluecrop	fruiting	2	14	0.100-0.101	EC	Foliar directed spray	36	Mature	Fruit	Hand	Not Provided
Burlington / WA/ 1999	12	Blueberry / Toro	fruiting	2	14	0.097-0.100	EC	Foliar directed spray	39-40	Mature	Fruit	Hand	Not Provided

[a] No tank mixes or adjuvants were used.

Comments: The field trials adequately represented the proposed use directions.

7. Site Specific Information

LOCATION (city/state)/ YEAR	FARMING PRACTICES			SOIL CHARACTERISTICS				
	CULTIVATION/ IRRIGATION*	FERTILIZER	MAINTENANCE CHEMICALS/RATE/TIMING	TYPE	% Organic Matter (OM)	pH	Cation Exchange Capacity (CEC)	WEATHER DATA** (T°C, rainfall)
Jonesboro/ ME/ 1999	Common cultural practices used to maintain plants	Maintenance chemicals and fertilizer used did not effect residue data		"sandy loam/loamy sand"	NR	4.8	Not provided	Unusually hot and dry during the blueberry growing season.
Fennville/ MI/ 1999	Common cultural practices used to maintain plants	Maintenance chemicals and fertilizer used did not effect residue data		"sandy loam"	3	5.0	Not provided	Air temperature and precipitation within normal range vs. historical weather data.
Fennville/ MI/ 1999	Common cultural practices used to maintain plants	Maintenance chemicals and fertilizer used did not effect residue data		"sandy loam"	3.6	4.7	Not provided	Air temperature and precipitation within normal range vs. historical weather data.
Fennville/ MI/ 1999	Common cultural practices used to maintain plants	Maintenance chemicals and fertilizer used did not effect residue data		"sand"	2.8	4.7	Not provided	Air temperature and precipitation within normal range vs. historical weather data.
Castle Hayne / NC/ 1999	Common cultural practices used to maintain plants	Maintenance chemicals and fertilizer used did not effect residue data		"sand"	5.6	NR	Not provided	Air temperature and precipitation within normal range vs. historical weather data.
Castle Hayne / NC/ 1999	Common cultural practices used to maintain plants	Maintenance chemicals and fertilizer used did not effect residue data		"sand"	5.6	NR	Not provided	Air temperature and precipitation within normal range vs. historical weather data.
Chatsworth/ NJ/ 1999	Common cultural practices used to maintain plants	Maintenance chemicals and fertilizer used did not effect residue data		"sand"	3.1	4.1	Not provided	Unusually hot and dry during the blueberry growing season.
Burlington / WA/ 1999	Common cultural practices used to maintain plants	Maintenance chemicals and fertilizer used did not effect residue data		"loam"	4.3	4.7	Not provided	Air temperature and precipitation within normal range vs. historical weather data.

II. RESULTS

TABLE 1. Residue Data Summary from Crop Field Trials

Location (city, state)/ Year	Crop/ Variety	Commodity/ Portion analysed	Formulation	Application[a]					PHI (days)	Pyriproxyfen Residues (mg/kg)
				Rate (lbs ai/A)	# of Appl.	Interval Between Appls. (days)	Total Rate (lbs ai/A)	Percent of Max Rate		
Jonesboro/ ME/ 1999	Blueberry/ Wild lowbush	Mature Fruit	EC	0.098 - 0.099	2	15	0.197	99	7	0.52, 0.60
Fennville/ MI/ 1999	Blueberry/ Rubel	Mature Fruit	EC	0.100 - 0.102	2	14	0.202	101	7	0.36, 0.45
Fennville/ MI/ 1999	Blueberry/ Rubel	Mature Fruit	EC	0.100 - 0.102	2	14	0.202	101	7	0.22, 0.15
Fennville/ MI/ 1999	Blueberry/ Rubel	Mature Fruit	EC	0.099 - 0.100	2	13	0.199	100	7	**0.59,0.64**
Castle Hayne/ NC/ 1999	Blueberry/ Croatan	Mature Fruit	EC	0.099	2	14	0.199	100	7	0.40, 0.48
Castle Hayne/ NC/ 1999	Blueberry/ Croatan	Mature Fruit	EC	0.099	2	15	0.198	99	6	0.33, 0.30
Chatsworth/ NJ/ 1999	Blueberry/ Bluecrop	Mature Fruit	EC	0.100 - 0.101	2	14	0.201	100	2	0.19, 0.14
									7	0.14, 0.15
									10	0.22, 0.10
									14	0.08, 0.07
									21	0.03, 0.07
Burlington / WA/ 1999	Blueberry/ Toro	Mature Fruit	EC	0.97 - 0.100	2	14	0.197	99	8	0.29, 0.23

[a] The maximum proposed application rate for pyriproxyfen to blueberry is 0.10 lb ai/A, or 0.20 lb ai/A/season.

Comments:
Two foliar applications of pyriproxyfen (0.86 lb/gal EC) at 0.098-0.102 lb ai/A/application were made to blueberry plants beginning at the small fruit stage for a total of 0.197-0.202 lb ai/A/season (0.985x to 1.01x the maximum proposed rate), at RTIs of 13-15 days. Mature blueberry fruit was harvested by hand 6-8 days after the last application.

The residue data are adequate and support a tolerance level of 1.0 ppm for pyriproxyfen residues in/on blueberry and the related crops currant, elderberry, gooseberry, huckleberry, lingonberry, juneberry, and salal ["Reviewer's Guide and Summary of HED ChemSAC Approvals for Amending Commodity Definitions [40 CFR § 180.1(h)] and Crop Group/Subgroups [40 CFR § 180.41]", B. Schneider, 20-SEP-2001]. In eight trials conducted in ME, MI, NC, NJ, and WA, pyriproxyfen residues were 0.14-0.64 ppm in/on blueberry samples, with a mean of 0.37 ppm. No LOD was provided. Adequate sample calculations and chromatograms were submitted.

The maximum frozen storage interval from collection to analysis was 54 days for samples from the blueberry fruit field trials. Frozen storage stability are available supporting the field trial data.

As there are no Mexican, Canadian or Codex MRLs established for pyriproxyfen in/on blueberry, there are no compatibility issues to be reconciled.

III. CONCLUSIONS

In eight trials conducted in ME, MI, NC, NJ, and WA, pyriproxyfen residues were 0.14-0.64 ppm in/on 16 blueberry samples harvested 6-8 days following the last of two foliar applications of pyriproxyfen (0.86 lb/gal EC) at 0.098-0.102 lb ai/A for a total of 0.197-0.202 lb ai/A/season. The field trial mean residue (FTMaR) for blueberry was 0.37 ppm.

Based on the concurrent method recoveries, the GC/NPD method (Method RM-33P-1-3a) is adequate for collecting data on residues of pyriproxyfen in/on blueberry. The method LOQ is 0.02 ppm for blueberry; no interference was observed in representative chromatograms of control samples from the analysis of blueberry fruit.

The residue data are acceptable and support a tolerance level of 1.0 ppm for pyriproxyfen residues in/on the bushberry subgroup and lingonberry, juneberry, and salal ["Reviewer's Guide and Summary of HED ChemSAC Approvals for Amending Commodity Definitions [40 CFR § 180.1(h)] and Crop Group/Subgroups [40 CFR § 180.41]", B. Schneider, 20-SEP-2001]. There are no Mexican, Canadian or Codex MRLs established for pyriproxyfen in/on blueberry.

IV. STUDY DEFICIENCIES

There were no deficiencies that would have an impact on establishment of the proposed tolerance.

V. REFERENCES

DP Barcode:	D238190
Subject:	PP#7F04882. Pyriproxyfen in/on Pome Fruits and Walnuts. Evaluation of Residue Data and Analytical Methods.
From:	W. Donovan
To:	S. Lewis/J. Tavano
Dated:	12/7/98
MRID:	443295-05 through -12

DP Barcode:	D253836
Subject:	PP#8F05022. Pyriproxyfen in/on Citrus Fruits, Fruiting Vegetables, and Tree Nuts. Evaluation of Residue Data and Analytical Methods.
From:	W. Donovan
To:	S. Lewis/J. Tavano
Dated:	3/25/99
MRID:	44638301, 44630102 through 44630107

DP Barcode:	D257337
Subject:	PP#s 7F04882 and 8F05022. Pyriproxyfen in/on Citrus Fruits, Fruiting Vegetables, and Tree Nuts. Results of Petition Method Validation (PMV) Request.
From:	W. Donovan
To:	J. Tavano/ A. Layne
Dated:	7/1/99
MRID(s):	None

Subject:	Reviewer's Guide and Summary of HED ChemSAC Approvals for Amending Commodity Definitions [40 CFR §180.1(h)] and Crop Group/Subgroups [40 CFR § 180.41]
From:	B. Schneider
To:	W. Hazel and H. Jamerson
Dated:	9/20/01
MRID(s):	None

cc: W. Donovan
RDI: RAB1 Chemists (29-NOV-2001)
W.H. Donovan:806R:CM#2:(703)305-7330:7509C:RAB1